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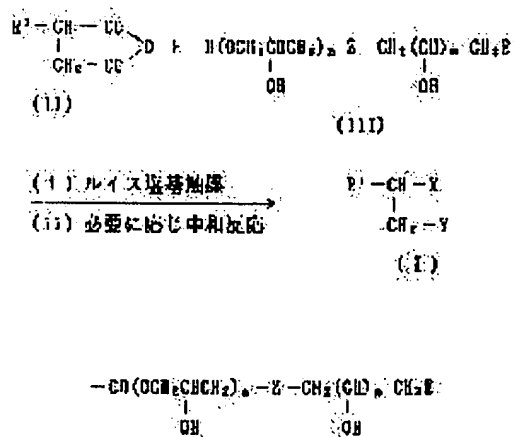
YOKOTA YUKINAGA

(54) **ALKYL-OR ALKENYLSUCCINIC ACID DERIVATIVE, ITS PRODUCTION AND SURFACTANT COMPOSED OF THE SAME COMPOUND**

(57)Abstract:

PURPOSE: To provide a new alkyl- or alkenylsuccinic acid derivative showing an excellent surfactant activity, mile to the skin, excellent in hard-water resistance and biodegradability and showing an excellent detergency to stains of a textile even in a form of a liquid detergent.

CONSTITUTION: A compound of formula I [R1 is 8-18C



straight or branched chain alkyl or alkenyl; one of X and Y is formula IV and the other is COOM; (n) is number of 0-10; Z is NR₂ (in this case; (m) is 4; E is OH and (n) is 0), O (in this case; (m) is 1; E is OH, SO₁M, formula V, OSO₃M, O(CH₂)_aCOOM; R₂ is 1-4C alkyl which may have OH; (a) is 1, 2 or 3; M is H, alkaline metal, ammonium, 2-12C alkanol-ammonium or 1-12C alkylammonium], e.g. 1-octenylsuccinic acid tetraglycerol ester. The above-mentioned compound can be synthesized from economical and readily available raw materials by reacting a compound of formula II with a compound of formula III in the presence of a Lewis base catalyst and subsequently neutralizing the reaction product, as necessary.

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[Date of extinction of right]



特 許 願 (二) 後 記 号 な し

昭和49年8月26日

特許庁長官 斎藤 英雄 殿

1. 発明の名称 FM通信方式

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5. 添付書類の目録

- | | |
|-------------|-----|
| (1) 明 細 書 | 1 通 |
| (2) 図 面 | 1 通 |
| (3) 委 任 状 | 1 通 |
| (4) 願 書 副 本 | 1 通 |

明 細 書

1. 発明の名称 FM通信方式

2. 特許請求の範囲

入力信号が存在するときのみ、該入力信号をFM変調して送信するFM通信方式に於いて、前記入力信号の検出遅れ時間以上の遅延時間を有する遅延回路を介して前記入力信号をFM変調器に加え、且つ前記入力信号の検出により前記FM変調器を動作させて、前記遅延回路による遅延時間と前記入力信号の検出遅れ時間との差の時間に無変調信号を送出することを特徴とするFM通信方式。

3. 発明の詳細な説明

本発明はFM通信方式、特に衛星通信に於けるBOPD方式を適用したFM通信方式に関するものである。

現在衛星通信にはBPADB方式が実用化されようとしており、この方式と同様な運用形式でデータ通信を取扱いBOPD方式も開発されている。これらの方式に於いては、その変調方式としてPBK

① 日本国特許庁 公開特許公報

①特開昭 51-25014

④公開日 昭51.(1976) 3.1

②特願昭 49-97646

②出願日 昭49.(1974) 8.26

審査請求 未請求 (全3頁)

庁内整理番号 7343 53

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96(7)A1

98(5)E0

96(7)B0

⑤ Int.Cl²

H04B 7/165

H04B 1/00

H03D 3/00

H04B 1/02

方式が採用されており、電話等の情報休止部分の電波の送信を停止して衛星の電力の有効利用を図っている。又電話通信が主として行なわれることを考えると、変調方式としてはFM変調方式を採用することも可能である。この場合、音声の所定時間以上の休止の場合に電波の送信が停止されるので、受信側においては、再び入力信号が復調器に加えられたとき、速かに復調信号が取出せるようにすることが要求される。

BPADB方式やBOPD方式のPBK波に於いては、バーストの先頭に同期用パターンが付加されており、その同期用パターンによりPBK波復調用の搬送波再生回路やクロック再生回路の同期確立が早急に行なわれるようになっている。FM変調方式を採用した場合にもバーストの先頭部に無変調信号を付加すれば前述のPBK波に対する場合と同様に同期確立所要時間を短くすることができ

本発明は前述の如きFM変調方式を採用し、バーストの先頭部に無変調信号を自動的に付加して

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通信を行なう方式を提供することを目的とするものである。以下実施例について詳細に説明する。

第1図は本発明の実施例のブロック図、第2図は動作説明波形図で、第1図の各部に於ける信号a～eは第2図のa～eに示されるものとなる。即ち音声信号aは音声検出器VDETと遅延回路DLとに加えられ、遅延回路DLにより例えば10msの遅延時間t1遅れた信号bがFM変調器FM MODに加えられる。又音声検出器VDETは、例えば最大7msの時間t2で音声信号aを検出し得るものであり、その検出信号cはハングオーバー回路HODに加えられ、検出信号cが“0”、即ち音声信号aが休止状態となつたときも、例えば150msの時間t4だけ信号dをFM変調器FM MODに加えるものである。このFM変調器FM MODは周波数シンセサイザ等を含むもので、信号dによつて動作開始して搬送波の送出が行なわれ、遅延された音声信号bの入力によつてFM変調が行なわれて、そのFM変調器FM MODの出力はeで示すものとなり、送信インタフェース回路IFTを介してアップコンバータ等

に送られ、所定の周波数の電波となつて送信される。この場合、第2図より明らかなように、FM変調器FM MODの出力eは、 $t_1 - t_2 = t_3$ の時間だけ無変調信号となる。この時間t3は前述の如く $t_1 = 10\text{ms}$ 、 $t_2 = 7\text{ms}$ とすると、 3ms となり、受信側の復調器の位相同期ループ回路の同期確立を、この3msの無変調信号によつて行ない、それにより確実に音声信号の復調を行なわせることができる。

なおハングオーバー時間t4は、音声信号が瞬断した場合にも搬送波送出を停止することがないようにする為に設定されており、前述の如く通常150ms程度とする。従つて遅延された音声信号bが休止状態となつた後、FM変調器FM MODからは時間t5(=150ms)だけ無変調信号が送出されることになる。

一方受信信号はダウンコンバータから受信インタフェース回路IFRを介してFM復調器FM DEMに加えられる。このFM復調器FM DEMには前述

の如く位相同期ループ回路が含まれており、バーストの先頭部の無変調信号により同期確立が行なわれて復調され、増幅器AMP等を経て出力される。

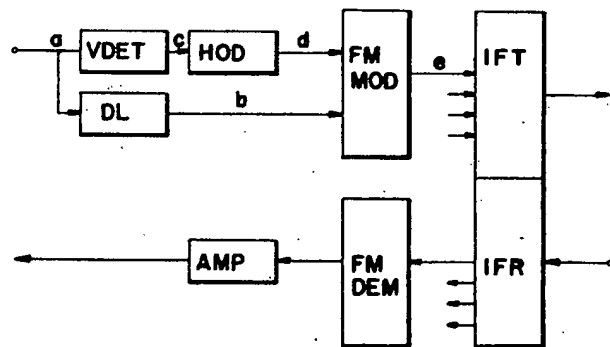
以上説明したように、本発明は、音声信号の検出遅れ時間よりも大きな遅延時間を有する遅延回路を介して音声信号をFM変調器に加え、このFM変調器を音声信号検出によつて動作させることにより、バーストの先頭部に無変調信号を付加するものであり、それによつて音声信号が存在するときのみ電波を送出するSOPPO方式に於ける復調器の位相同期ループ回路の同期確立を速に行なわせることができるものである。

4. 図面の簡単な説明

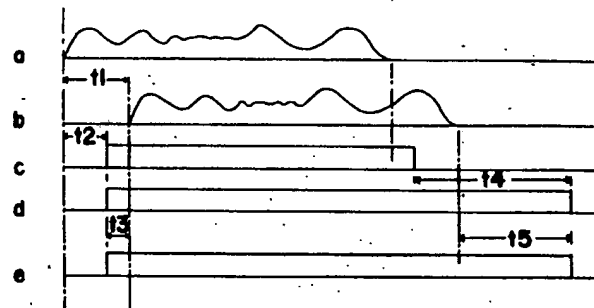
第1図は本発明の実施例のブロック図、第2図はその動作説明図である。

VDETは音声検出器、HODはハングオーバー回路、DLは遅延回路、FM MODはFM変調器、IFTは送信インタフェース回路、IFRは受信インタフェース回路、FM DEMはFM復調器である。

第 1 図



第 2 図



6. 前記以外の発明者および代理人

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CLAIMS

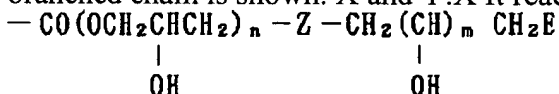
[Claim(s)]

[Claim 1] The alkyl or the alkenyl succinic-acid derivative expressed with a general formula (I).

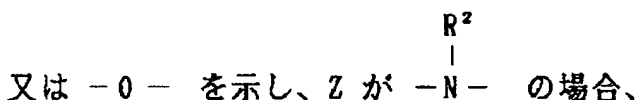
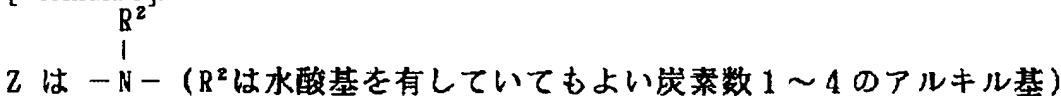
[Formula 1]



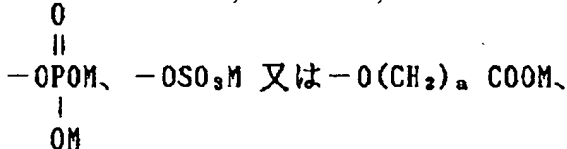
The inside of [type, R1: The alkyl group or alkenyl radical of carbon numbers 8-18 of a straight chain or branched chain is shown. X and Y: X It reaches. One side among Y is [Formula 2].



An example and another side show -COOM. Here, it is n. The number of 0-10 is shown and it is [Formula 3].



4 and E show OH, n shows 0, and m is Z. It is m when it is -O-. 1 E OH, SO₃M, [Formula 4]



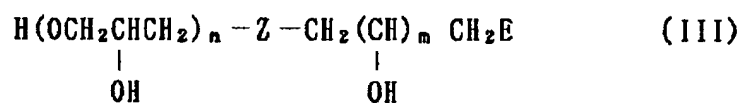
n The number of ** 0-10 is shown. However, a The number of 1-3 is shown and M shows H, alkali metal, ammonium, alkanol ammonium with 2-12 total carbon, or alkylammonium with 1-12 total carbon.]

[Claim 2] The manufacture approach of the alkyl which is characterized by making the alkyl or the alkenyl succinic-acid anhydride expressed with the following general formula (II), and the compound expressed with the following general formula (III) react to the bottom of Lewis base existence and which is expressed with a general formula (I) according to claim 1, or an alkenyl succinic-acid derivative.

[Formula 5]



(式中、R¹は前記の意味を示す。)



(式中、Z、E、m及びnは前記の意味を示す。)

[Claim 3] The surfactant which consists of alkyl according to claim 1 or an alkenyl succinic-acid derivative.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to new alkyl or an alkenyl succinic-acid derivative, and its manufacture approach. Furthermore, it is related with the multi-hydrophilic-group mold activator which has an amide or an ester group useful as a detailed low stimulative surfactant, and its manufacture approach.

[0002]

[Description of the Prior Art] A surfactant tends to consist of a non-dense water space and a hydrophilic part, tends to distribute water, an oil and water, dirt and water, a metal, etc. efficiently, and tends to obtain various industrial and homely functions using these emulsification, humidity, and a distributed condition. In washing especially by distribution of dirt, the fall of foamability and a detergency becomes a problem in response to the effect of polyvalent metal ion, such as calcium in hard water, magnesium, iron, and aluminum. Although it excels in foamability and stability in hard water, and it is mild, and biodegradability is good and a pollution-free thing is desired to the skin when using it especially for a shampoo and a body cleaning agent, what may fully be satisfied is not offered. Moreover, although various constituents are needed for obtaining a detergency, without being influenced of polyvalent metal ion also with a liquid garments detergent, the satisfactory product is not necessarily developed.

[0003]

[Means for Solving the Problem] A header and this invention were completed for it having the detergency which it is mild to the skin while having the surface activity ability excellent in the compound expressed with the following general formula (I) as a result of this invention person's examining this situation wholeheartedly, and it excels in stability in hard water, and whose biodegradability is good, and was excellent also in the liquid type to the dirt of fiber, and being able to compound from an available raw material cheaply further moreover easily. That is, this invention offers the surfactant which becomes the new alkyl expressed with a general formula (I) or an alkenyl succinic-acid derivative and its manufacture approach, and a list from this alkyl or an alkenyl succinic-acid derivative.

[0004]

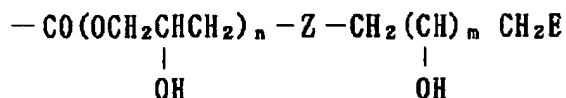
[Formula 6]



[0005] The inside of [type, R1: The alkyl group or alkenyl radical of carbon numbers 8-18 of a straight chain or branched chain is shown.

[0006] X and Y: X It reaches. One side among Y is [0007].

[Formula 7]



[0008] An example and another side show -COOM. Here, it is n. The number of 0-10 is shown and it is [0009].

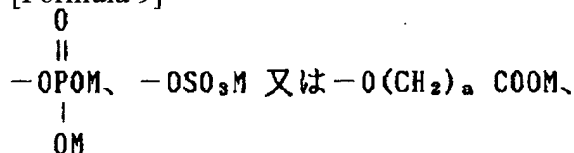
[Formula 8]

Z は $-\text{N}-$ (R^2 は水酸基を有していてもよい炭素数1~4のアルキル基)

又は $-\text{O}-$ を示し、Z が $-\underset{\text{R}^2}{\text{N}}-$ の場合、

[0010] 4 and E show OH, n shows 0, and m is Z. It is m when it is -O-. 1 E OH, SO₃M, [0011]

[Formula 9]

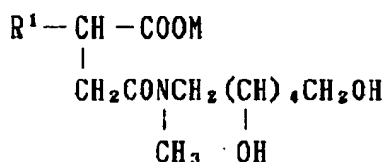
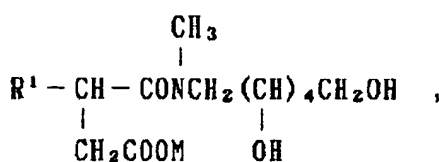
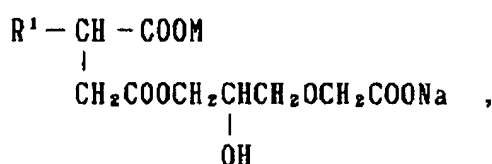
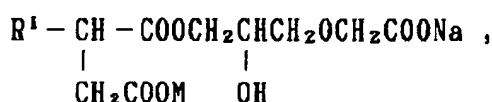
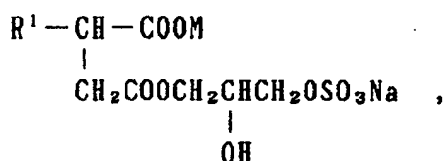
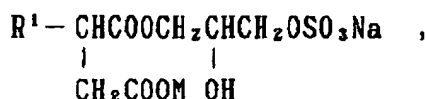
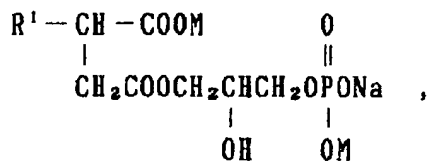
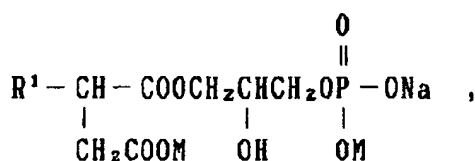
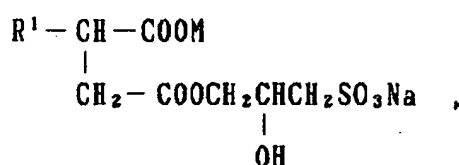
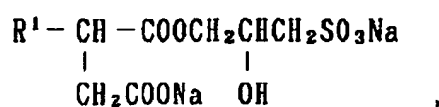


[0012] n The number of ** 0-10 is shown. However, a The number of 1-3 is shown and it is M. H, alkali metal, ammonium, alkanol ammonium with 2-12 total carbon, or alkylammonium with 1-12 total carbon is shown.] This invention is explained to a detail below. In this invention compound expressed with said general formula (I), alkenyl radicals, such as alkyl groups, such as an octyl radical, a nonyl radical, a decyl group, an undecyl radical, a lauryl radical, the Millis Chill radical, a palmityl radical, and a stearyl radical, an octenyl group, a NONENIRU radical, a decenyl radical, an undecenyl radical, a dodecenyl radical, an arachidyl radical, an oleyl radical, and a RINORU radical, be mentioned as the alkyl group or alkenyl radical of carbon numbers 8-18 of the straight chain shown by R1, or branched chain

[0013] Moreover, M Although a hydrogen atom, alkali metal, ammonium, alkanol ammonium with 2-12 total carbon, or alkylammonium with 1-12 total carbon is shown, as an example of alkanol ammonium with 2-12 total carbon, mono-ethanol ammonium, JIETA Norian ammonium, triethanol ammonium, etc. are mentioned, and methylammonium, dimethylammonium, trimethylammonium, tetramethylammonium, triethyl ammonium, etc. are mentioned as an example of alkylammonium with 1-12 total carbon here. As an example of this invention compound expressed with said general formula (I), the compound expressed with the following formula, for example is mentioned. In addition, the inside of a series of formulas, and R1 and M Above semantics is shown.

[0014]

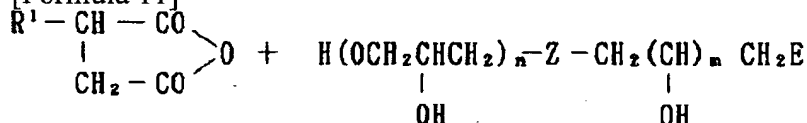
[Formula 10]



[0015] this invention compound (I) is manufactured according to a degree type.

[0016]

[Formula 11]

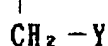
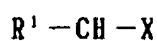


(II)

(III)

(i) ルイス塩基触媒

(ii) 必要に応じ中和反応



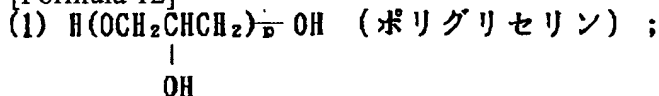
(I)

[0017] (R1, X, Y, Z, E, m, and n show above semantics among a formula.) the compound expressed with the general formula (II) which is starting material -- for example, it can use as the inside of an inert gas ambient atmosphere by the well-known approach, and an alpha olefin and a maleic anhydride can be used as an alkenyl succinic-acid anhydride by the en reaction. Moreover, after making an alpha olefin and succinic-acid diester into alkyl succinate by radical reaction, it can consider as an alkyl succinic-acid anhydride by anhydride-izing. In addition, an alkyl succinic-acid anhydride is obtained also by carrying out hydrogenation of the alkenyl succinic-acid anhydride. As a compound expressed with a general

formula (III), it is (1) of the following, for example. - (6) A compound is mentioned.

[0018]

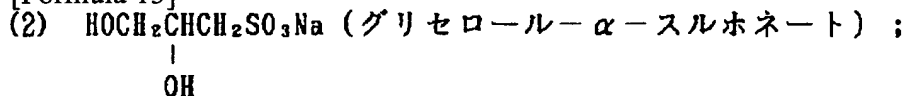
[Formula 12]



[0019] About [(p)2-10] polyglycerin can be obtained whenever [average condensation] by condensing a glycerol at an elevated temperature under a base catalyst. Moreover, the same polyglycerin can be obtained also by the polymerization of glycidol.

[0020]

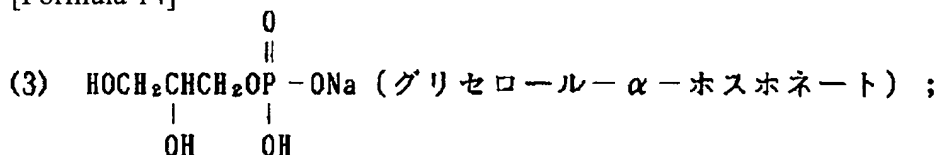
[Formula 13]



[0021] Glycerol-alpha-sulfonate can be obtained by making glycerol-alpha monochlorohydrin and Na2SO3 react. Moreover, glycerol-alpha-sulfonate can be similarly obtained by hydrolyzing epichlorohydrin and NaHSO3 after a ring closure in NaOH after reacting.

[0022]

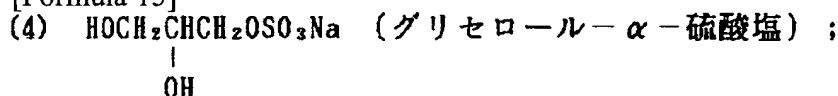
[Formula 14]



[0023] Glycidol and NaH2PO4 Glycerol-alpha-phosphonate can be obtained by making it react.

[0024]

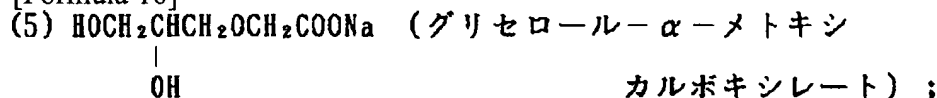
[Formula 15]



[0025] a glycerol -- H2NSO3H etc. -- a glycerol-alpha-sulfate can be obtained by making it react with a sulfation reagent. In addition, the glycerol-alpha-sulfate of a high grade can be obtained more by using the 2 and 2-dimethyl -1 and a 3-dioxolane-4-methanol instead of a glycerol.

[0026]

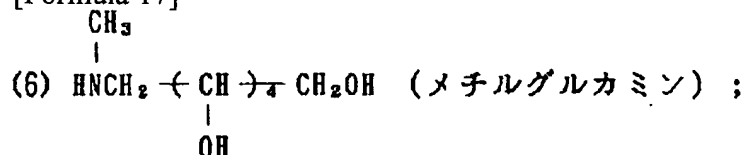
[Formula 16]



[0027] Glycerol-alpha-methoxy carboxylate can be obtained by making a glycerol into a catalyst and making NaOH it react with ClCH2COONa. In addition, the glycerol-alpha-methoxy carboxylate of a high grade can be obtained more by use of the 2 and 2-dimethyl -1 and a 3-dioxolane-4-methanol.

[0028]

[Formula 17]



[0029] They are a glucose and monomethylamine Pd/C It can consider as a catalyst and a methyl glucamine can be obtained by carrying out hydrogenation after a reaction.

[0030] The reaction of a compound (II) and a compound (III) distributes or dissolves the reaction by package preparation, or a compound (III) into a solvent under Lewis base catalyst existence, and after a temperature up, a compound (II) is conversely dissolved into a solvent and it is performed after a temperature up by the approach of trickling a compound (II), or the approach to which add a compound (III) and it is made to react. Setting to the invention in this application, the mole ratio of a compound (II) and a compound (III) is usually compound (II) / compound (III) = 1/0.8 - 1/1.3. It is the range. As a catalyst used in this reaction, a Lewis base is desirable, and phosphoric ester compounds, such as ether compounds, such as nitrogen-containing compounds, such as triethylamine, a trimethylamine, a pyridine, pyrrolidine, a lutidine, and dimethylformamide, and dioxane, and tributyl phosphate, etc. are mentioned as a Lewis base. When using a reaction catalyst, the amount used receives a compound (II). It is more than 0.1 mol %.

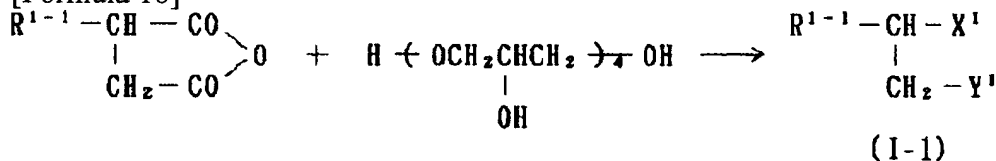
[0031] Reaction temperature can complete 50-150 °C in about 1 - 46 hours, and reaction time can complete a reaction. Moreover, as a solvent used in this reaction, inert solvents, such as ether, such as glycol ether and cyclic ether, are mentioned. In addition, the Lewis base used as a catalyst can also be used as a solvent. After reaction termination, a solvent and a catalyst are removed from a resultant by well-known technique, such as reduced pressure drying, and the compound (I) of this invention is obtained. Although this compound (I) can also be used as it is, it refines by the well-known approach using an organic solvent etc., and can be used, being able to raise the purity of a compound (I). this invention compound (I) can be easily dissolved in water, and can be made into a neutralization salt with a basic compound. In this case, as a base used, alkanolamines, such as low-grade alkylamines, such as alkali-metal hydroxides and ammonia, such as a sodium hydroxide and a potassium hydroxide, and triethylamine, and monoethanolamine, etc. are mentioned.

[0032]

[Example] Hereafter, although an example explains this invention to a detail further, this invention is not limited to these examples.

[0033] Composition of an example 11-OKUTE nil succinic acid and tetra-glycerol ester (I-1) [0034]

[Formula 18]



〔式中、R¹⁻¹：1-オクテニル基を示す。〕

X¹, Y¹：一方は $-\text{CO}-\text{C}(\text{OH})-\text{CH}_2-\text{CH}_2-\text{OH}$ を示し、
 OH

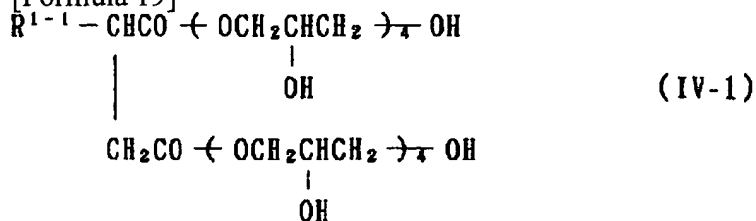
他方は、 $-\text{COONa}$ を示す。〕

[0035] 500ml equipped with the agitator, the cooling pipe, and the thermometer Tetra-glycerol compounded in the three opening flask by the condensation of 80g (0.38 mols) of 1-OKUTE nil succinic-acid anhydrides, and a glycerol 143.4g (0.46 mols) is added -- it agitated at 100 degree C for 6 hours. Ethyl acetate after washing a product by the hexane It is made to dissolve in 500ml and is brine. It washed 3 times by 200ml. It is a methanol about the residue which dried the ethyl-acetate layer by Na₂SO₄, and was obtained by distilling off ethyl acetate in the rotary evaporator. It dissolved in 500ml, and this molar quantity addition of the NaOH water solution was carried out to the carboxyl group 48%, and it neutralized. In addition, in order to prevent decomposition of ester in the case of neutralization, pH was made or less into nine and temperature was made into 20 degrees C or less. The obtained

solution was condensed, it dried and 1166g of dry matters was obtained. As a result of high performance chromatography's analyzing this dry matter, the compound expressed with the above-mentioned formula (I-1) and the compound expressed with the following type (IV-1) checked generating by the weight ratio at 95/5.

[0036]

[Formula 19]



[0037] (R1-1 shows above semantics among a formula.)

It is shown in the compound and the fats-and-oils analysis table 1 expressed with a <analysis result> type (I-1).

[0038]

[Table 1]

	AV (mg-KOH/g)	SV (mg-KOH/g)	OHV (mg-KOH/g)
測定値	- 99.6	210.2	510.8
理論値	-102.7	205.4	513.3

[0039] The unit of AV, SV, and OHV is [the following and] mg-KOH/g.

- 3700-3000cm⁻¹ of IR analysis: OH stretching vibration 1728 cm⁻¹ : C=O Stretching vibration (ester)
1575 cm⁻¹ : C=O It is shown in the compound and the fats-and-oils analysis table 2 expressed with a stretching-vibration (COONa) type (IV-1).

[0040]

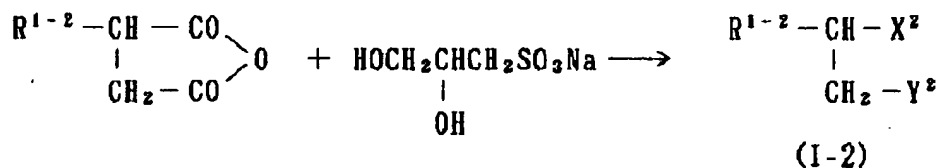
[Table 2]

	SV	OHV
測定値	131.4	680.2
理論値	136.7	683.3

[0041] - 3700-3000cm⁻¹ of IR analysis: OH stretching vibration 1725 cm⁻¹ : C=O Stretching vibration (ester)

Composition of an example 21-dodecenyl succinic acid and hydroxypropyl sulfonate ester (I-2) [0042]

[Formula 20]



(式中、 R^{1-2} : 1-ドデセニル基を示す。

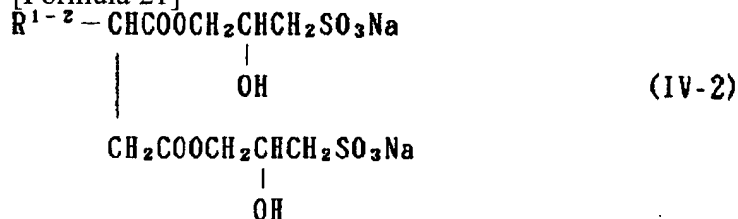
X^2, Y^2 : 一方は $-COOCH_2CHCH_2SO_3Na$ を示し、
|
OH

他方は、 $-COONa$ を示す。)

[0043] 399.6g [of 1-dodecenyl succinic-acid anhydrides] (1.5 mol) and glycerol-alpha-sulfonate 267.2g (1.5 mol) and pyridine 15g are added to three 1 liter-capacity opening flask equipped with the agitator, the cooling pipe, and the thermometer -- it agitated at 130 degree C for 16 hours. After washing a product by the hexane, it was made to dissolve in ethanol and insoluble matter was separated. It is a methanol about the residue obtained by distilling off ethanol in a rotary evaporator. It dissolved in 500ml and neutralized by carrying out equimolecular amount addition of the NaOH water solution to a carboxyl group 48%. In addition, in order to prevent decomposition of ester in the case of neutralization, pH was made or less into nine and temperature was made into 20 degrees C or less. Dry matter after condensing the obtained solution and drying 407g was obtained. As a result of high performance chromatography's analyzing this dry matter, the compound expressed with the above-mentioned formula (I-2) and the compound expressed with the following type (IV-2) checked generating by the weight ratio at 89/11.

[0044]

[Formula 21]



[0045] (R1-2 show above semantics among a formula.)

It is shown in the compound and the fats-and-oils analysis table 3 expressed with a <analysis result> type (I-2).

[0046]

[Table 3]

	AV	SV	OHV
測定値	-122.2	243.0	123.1
理論値	-120.3	240.6	120.3

[0047] - 3700-3000cm-1 of IR analysis: OH stretching vibration 1726 cm-1 : C=O Stretching vibration (ester)

1576 cm-1 : C=O Stretching vibration 1195 (COONa) cm-1 : S=O It is shown in the compound and the fats-and-oils analysis table 4 expressed with a stretching-vibration type (IV-2).

[0048]

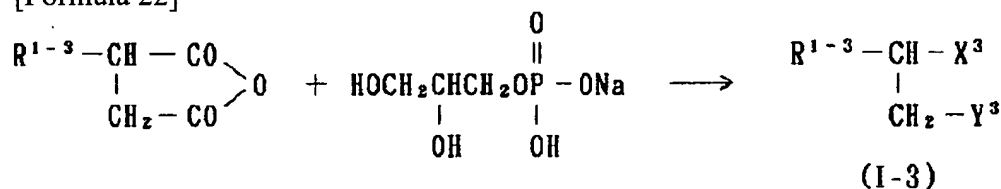
[Table 4]

	SV	OHV
測定値	183.9	188.4
理論値	185.6	185.6

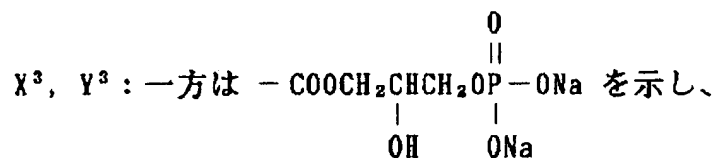
[0049] Composition of an example 31-stearyl succinic acid and hydroxypropyl phosphate ester (I-3)

[0050]

[Formula 22]



〔式中、 R^{1-3} ：1-ステアリル基を示す。〕

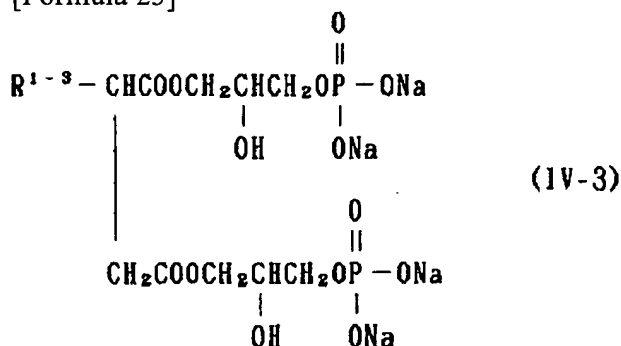


他方は、 $-COONa$ を示す。〕

[0051] 500ml equipped with the agitator, the cooling pipe, and the thermometer It is a stearyl succinic-acid anhydride to a three ** opening flask. 105.8g (0.3 mol), glycerol-alpha-phosphate 69.9g (0.4 mols), pyridine 5g, and DMF 100 g were taught, and it agitated at 80 degrees C for 36 hours. After distilling off DMF and a pyridine in the rotary evaporator and washing residue by the hexane, it was made to dissolve in an acetone and insoluble matter was separated. It is a methanol about the residue obtained by distilling off ethanol in a rotary evaporator. It dissolved in 200ml and neutralized by carrying out two-mol equivalent addition of the NaOH water solution to a product 48%. In addition, in order to prevent decomposition of ester in this case, 8 or less and temperature made pH 20 degrees C or less. After condensing the obtained solution and drying, 88.6g of dry matters was obtained. As a result of high performance chromatography's analyzing this dry matter, the compound expressed with the above-mentioned formula (I-3) and the compound expressed with the following type (IV-3) checked generating by the weight ratios 97/3.

[0052]

[Formula 23]



[0053] (R1-3 show above semantics among a formula.)

It is shown in the compound and the fats-and-oils analysis table 5 expressed with a <analysis result>

type (I-3).

[0054]

[Table 5]

	AV	SV	OHV
測定値	-287.1	377.4	92.3
理論値	-285.0	380.0	95.0

[0055] - 3650-3000cm⁻¹ of IR analysis: OH stretching vibration 1724 cm⁻¹ : C=O Stretching vibration (ester)

1577 cm⁻¹ : C=O Stretching vibration 1256 (COONa) cm⁻¹ : P=O It is shown in the compound and the fats-and-oils analysis table 6 expressed with a stretching-vibration type (IV-3).

[0056]

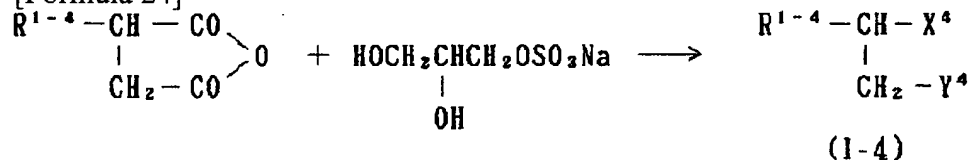
[Table 6]

	SV	OHV
測定値	567.3	193.4
理論値	570.0	190.0

[0057] - 3650-3000cm⁻¹ of IR analysis: OH stretching vibration 1726 cm⁻¹ : C=O Stretching vibration (ester)

1255 Cm⁻¹ : P=O Composition of Stretching-Vibration Example 41-Decenyl Succinic Acid and Hydroxypropyl Sulfate Ester (I-4) [0058]

[Formula 24]



[式中、R¹⁻⁴ : 1-デセニル基を示す。

X⁴, Y⁴ : 一方は $-COOCH_2CHCH_2OSO_3Na$ を示し、
OH

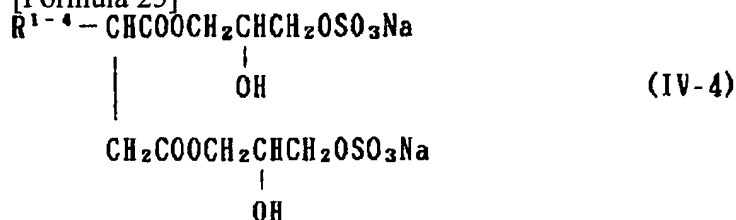
他方は、-COONa を示す。]

[0059] It is ethylene glycol wood ether as glycerol-alpha-sulfate 58.2g (0.3 mol), 71.5g [of decenyl succinic-acid anhydrides] (0.3 mol), and pyridine 5g, and a solvent. 500ml equipped with the agitator, the cooling pipe, and the thermometer for 200g It taught the three ** opening flask and flowed back for 20 hours. After distilling off the solvent in the rotary evaporator and washing the obtained product by the hexane, it was made to dissolve by ethanol and insoluble matter was separated. It is a methanol about the residue obtained by distilling off ethanol in a rotary evaporator. It dissolved in 200ml and neutralized by carrying out equimolecular amount addition of the NaOH water solution to a carboxyl group 48%. In addition, in order to prevent decomposition of ester in the case of neutralization, pH was made or less into nine and temperature was made into 20 degrees C or less. After condensing the obtained solution and drying, 97.4g of dry matters was obtained. As a result of high performance chromatography's analyzing this dry matter, the compound expressed with the above-mentioned formula (I-4) and the compound expressed with the following type (IV-4) checked generating by the weight

ratios 93/7.

[0060]

[Formula 25]



[0061] (R1-4 show above semantics among a formula.)

It is shown in the compound and the fats-and-oils analysis table 7 expressed with a <analysis result> type (I-4).

[0062]

[Table 7]

	AV	SV	OHV
測定値	-125.1	243.7	122.9
理論値	-123.5	246.9	123.5

[0063] - 3700-3000cm⁻¹ of IR analysis: OH stretching vibration 1725 cm⁻¹ : C=O Stretching vibration (ester)

1580 cm⁻¹ : C=O Stretching vibration 1185 (COONa) cm⁻¹ : S=O It is shown in the compound and the fats-and-oils analysis table 8 expressed with a stretching-vibration type (IV-4).

[0064]

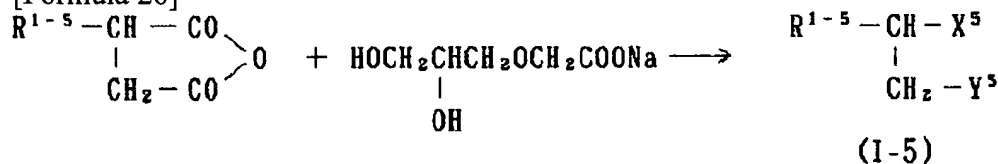
[Table 8]

	SV	OHV
測定値	182.7	187.3
理論値	184.4	184.4

[0065] - 3650-3000cm⁻¹ of IR analysis: OH stretching vibration 1723 cm⁻¹ : C=O Stretching vibration (ester)

1187 Cm⁻¹ : S=O Composition of Stretching-Vibration Example 5 Dodecyl Succinic Acid and Alpha-Carboxymethyl Glycerol Ester [0066]

[Formula 26]



〔式中、R¹⁻⁵ : ドデシル基を示す。〕

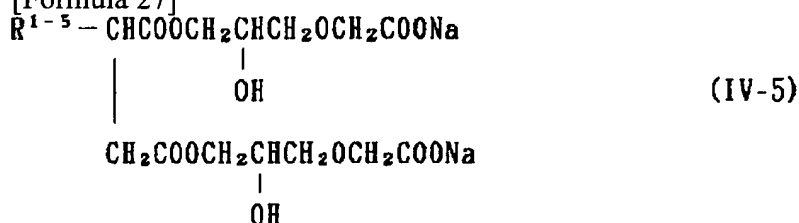
X⁵, Y⁵ : 一方は $-\text{COOCH}_2\text{CHCH}_2\text{OCH}_2\text{COONa}$ を示し、
 $\qquad \qquad \qquad |$
 $\qquad \qquad \qquad \text{OH}$

他方は、-COONa を示す。〕

[0067] Glycerol-alpha-carboxy methyl ether 51.6g (0.3 mols) and 500ml equipped with the agitator, the cooling pipe, and the thermometer for DMF 200ml It taught the three ** opening flask, and it heated, agitating. In the place where reflux started, 80.5g (0.3 mol) of dodecyl succinic-acid anhydrides was dropped over 1 hour. After continuing reflux for 18 hours, the solvent was distilled off in the rotary evaporator. After washing the obtained product by the hexane, it was made to dissolve by ethanol and insoluble matter was separated. It is a methanol about the residue obtained by distilling off ethanol in a rotary evaporator. It dissolved in 200ml and neutralized by carrying out equimolecular amount addition of the NaOH water solution to a carboxyl group 48%. In addition, in order to prevent decomposition of ester in the case of neutralization, pH was made or less into nine and temperature was made into 20 degrees C or less. Dry matter after condensing the obtained solution and drying 108.4g was obtained. As a result of high performance chromatography's analyzing this dry matter, the compound expressed with the above-mentioned formula (I-5) and the compound expressed with the following type (IV-5) checked generating by the weight ratios 93/7.

[0068]

[Formula 27]



[0069] (R1-5 show above semantics among a formula.)

It is shown in the compound and the fats-and-oils analysis table 9 expressed with a <analysis result> type (I-5).

[0070]

[Table 9]

	AV	SV	OHV
測定値	244.4	360.2	121.5
理論値	242.6	363.9	121.3

[0071] - 3600-3000cm⁻¹ of IR analysis: OH stretching vibration 1728 cm⁻¹ : C=O Stretching vibration (ester)

1565 cm⁻¹ : C=O It is shown in the compound and the fats-and-oils analysis table 10 expressed with a stretching-vibration (COONa) type (IV-5).

[0072]

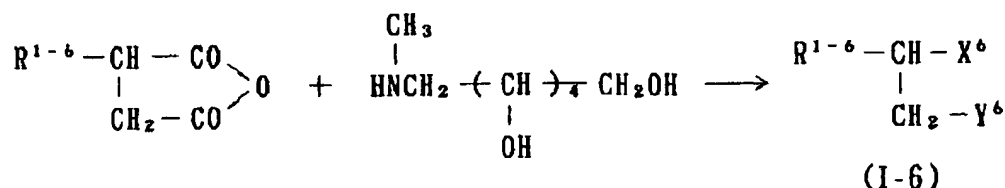
[Table 10]

	SV	OHV
測定値	375.4	190.8
理論値	377.4	188.7

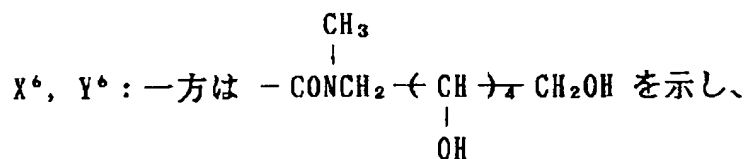
[0073] - 3650-3000cm⁻¹ of IR analysis: OH stretching vibration 1728 cm⁻¹ : C=O Stretching vibration (ester)

1567 Cm⁻¹ : C=O Composition of Stretching-Vibration (COONa) Example 61-OKUTE Nil Succinic Acid and Methyl Glucamine Amide [0074]

[Formula 28]



[式中、R¹⁻⁶：1-オクテニル基を示す。



他方は、-COOHを示す。]

[0075] 500ml equipped with the agitator, the cooling pipe, and the thermometer To a three ** opening flask, they are N-methyl glucamine 33.0g (0.17 mols) and a pyridine. The temperature up was carried out to 50 degrees C, adding and agitating 100g. 30.0g (0.14 mols) of 1-OKUTE nil succinic-acid anhydrides was added keeping at 50 degrees C, and it agitated at 60-degree-Cdegree C for 5 hours. it is made to dissolve in an acetone and insoluble, after ethyl acetate washes the product obtained by distilling off a pyridine in a rotary evaporator -- unreacted N-methyl glucamine was separated. After the silica gel column refined the residue obtained by distilling off an acetone in a rotary evaporator, 20.0g of products was obtained. As a result of high performance chromatography's analyzing this product, it checked that the compound expressed with the above-mentioned formula (I-6) was generating.

[0076] It is shown in the compound and the fats-and-oils analysis table 11 expressed with a <analysis result> type (I-6).

[0077]

[Table 11]

	AV	OHV
測定値	135.0	694.8
理論値	138.4	691.8

[0078] - 3700-3000cm⁻¹ of IR analysis: OH stretching vibration 1719 cm⁻¹ : C=O Stretching vibration (COOH)

1623 Cm⁻¹ : C=O Telescopic Motion and NH Deformation Vibration (CON -- <)

The example 1 (measurement of the foam formation force) of a trial

0.05g of compounds obtained in the examples 1-6 was dissolved in 25ml of ion exchange water, it put into the 250 ml cylinder with a graduation, and the amount of foam formation immediately after shaking 20 times was measured. A result is shown in Table 12.

[0079]

[Table 12]

化合物	I-1	I-2	I-3	I-4	I-5	I-6
起泡量 (ml)	240	190	120	200	150	180

化合物	IV-1	IV-2	IV-3	IV-4	IV-5
起泡量 (ml)	120	100	140	90	150

[0080] The example 2 (measurement of surface tension) of a trial

The compound obtained in the examples 1-6 reaches 1% of the weight. About the surface tension in the room temperature of a 0.1-% of the weight water solution, it is Willhelmy. It measured using the mold surface tension balance. A result is shown in Table 13.

[0081]

[Table 13]

化合物	表面張力 (dyn/cm)		化合物	表面張力 (dyn/cm)	
	1 重量%	0.1 重量%		1 重量%	0.1 重量%
I - 1	2 8	3 0	IV - 1	3 5	3 6
I - 2	3 1	3 1	IV - 2	4 6	4 7
I - 3	4 1	3 7	IV - 3	3 2	3 0
I - 4	4 0	4 0	IV - 4	4 3	4 5
I - 5	4 2	4 3	IV - 5	3 0	3 5
I - 6	3 3	3 5			

[0082]

[Effect of the Invention] It is a new molecular entity, and while it has the outstanding surface activity ability, to the skin, the compound of this invention expressed with the above-mentioned general formula (I) is mild, excellent in stability in hard water, and biodegradability is good, can have the detergency which was excellent also in the liquid type to the dirt of fiber, and it can compound it from an available raw material easily still more cheaply.

[Translation done.]